APR 1 1981

Item 830 H-15

NAS 1.60:1813

NASA Technical Paper 1813

COMPLETED

Surface Chemistry and Friction Behavior of the Silicon Carbide (0001) Surface at Temperatures to 1500° C

Kazuhisa Miyoshi and Donald H. Buckley

MARCH 1981

13

NASA

NASA Technical Paper 1813

Surface Chemistry and Friction Behavior of the Silicon Carbide (0001) Surface at Temperatures to 1500° C

Kazuhisa Miyoshi and Donald H. Buckley
Lewis Research Center
Cleveland, Ohio

NASA
National Aeronautics
and Space Administration

**Scientific and Technical
Information Branch**

1981

A

Summary

An investigation was conducted to examine the (0001) surface of silicon carbide at various temperatures to 1500° C by X-ray photoelectron and Auger electron spectroscopies. The friction behavior of a single-crystal silicon carbide surface in contact with polycrystalline iron was also examined. Surface analyses and sliding friction experiments were conducted in a vacuum system of 10^{-8} pascal. All friction experiments were conducted with loads to 0.2 newton, at a sliding velocity of 3×10^3 meter per minute, and in a vacuum of 10^{-8} pascal (10^{-10} torr).

The results of the investigation indicate a significant temperature influence on the surface and friction properties of silicon carbide. The principal contaminant on the as-received silicon carbide is carbon, which disappeared on heating to 400° C. Above 400° C graphite and carbide are the primary species, in addition to silicon. The coefficients of friction of polycrystalline iron sliding against a single-crystal silicon carbide (0001) surface were high at temperatures to 800° C. Similar coefficients of friction were obtained at room temperature on the silicon carbide that had been preheated at various temperatures up to 800° C. When the friction experiments were conducted at temperatures above 800° C or when the specimens were preheated to temperatures above 800° C, the coefficients of friction were dramatically lower. It has been determined that at 800° C, the silicon and carbide-type carbon are at a maximum intensity in the XPS spectra. Increasing temperatures above 800° C result in the concentration of the graphite increasing rapidly on the surface, whereas the concentrations of the carbide-type carbon and silicon decrease rapidly.

Introduction

Because silicon carbide has high strength and excellent oxidation, creep, and wear resistances, its applications include stable, high-temperature semiconductors, gas turbine blades, turbine ceramic seals, and as an abrasive for grinding (refs. 1 and 2). In grinding, the extremely small chip size and the high wheel speed involved produce instantaneous temperatures and pressures at the tip of the abrasive silicon carbide that are extremely high.

Further, the freshly formed surfaces of silicon carbide at high temperatures are highly reactive with metals (ref. 3). Neither the fundamentals of the surface chemistry involved nor the tribological properties of the silicon carbide at these conditions is clearly understood.

The present authors have conducted experimental research at elevated temperatures to gain an understanding of the surface chemistry and tribological properties of silicon carbide. The surfaces of silicon carbide preheated to 800° C are almost the same as that sputter-cleaned with argon ions at room temperature. Such clean surfaces reveal the main silicon and carbide-type carbon peak in the Auger spectra. The surfaces heated at 1500° C, however, reveal only a graphite-type carbon peak, the silicon peak being considerably diminished. The surfaces of the clean silicon carbide preheated at 800° C in sliding contact with iron gave a 1.5 to 3 times higher coefficient of friction than did the surface of silicon carbide preheated at 1500° C when friction experiments were conducted in the range of room to 800° C. Thus, the surface characteristics of silicon carbide are strongly affected by temperature (ref. 4).

The details of the surfaces, however, heated to 1500° C were not described in reference 4, except at the specific temperatures of 800° and 1500° C.

The objective of the present paper is to describe the surface chemistry of silicon carbide and the adhesion and friction behavior of silicon carbide sliding against iron at various elevated temperatures. Surfaces of silicon carbide were resistance-heated to 1500° C in a 10^{-8} pascal vacuum and were then analyzed at room temperature. The surface chemistry of silicon carbide crystals was analyzed by X-ray photoelectron spectroscopy (XPS), more popularly known as electron spectroscopy for chemical analysis (ESCA), as well as by Auger Electron Spectroscopy (AES). Though XPS and AES both provide a surface composition analysis, each technique has its advantages: AES offers superior spatial resolution and obtains results in a shorter time frame than XPS and is the preferred technique for rapid elemental analysis of specimens; XPS on the other hand is the preferred analysis technique when electron radiation damage is of concern, since X-ray excitation causes very little radiation damage to most specimens. It also provides more detailed chemical information

than AES. Sliding friction experiments were conducted with a single- crystal silicon carbide (0001) surface in contact with a polycrystalline iron surface. All friction experiments were conducted with loads to 0.2 newton, at a sliding velocity of 3×10^{-3} meter per minute, and in a 10^{-8} pascal (10^{-10} torr) vacuum.

Materials

The α single-crystal silicon carbide used in the experiment was a 99.9 percent pure compound of silicon and carbon. The unit cell of α-SiC is hexagonal with $a = 3.0817$ Å and $c = 15.1183$ Å and has 21 molecules per unit cell (refs. 5 and 6). The method of growth is by carbon arc. The (0001) plane was nearly parallel to the sliding surfaces examined herein. X-ray back reflection Laue photographs were taken to establish the exact bulk orientation of the crystal after it had been polished with a diamond powder (3 μm diam.) and then an aluminum oxide powder (1 μm diam.).

Specimens were within $\pm 2^\circ$ of the low index (0001) plane. The silicon carbide samples were in the form of flat platelets and had a mean surface area of about 70 millimeters or more. The roughness of the mirror-polished silicon carbide surfaces measured by surface profilometer was 0.1 micrometer for the maximum height of irregularities (R_{max}). The iron was polycrystalline, and 99.99 percent pure. The method of preparation was by electron beam zone refining. The radius of the iron pin specimen was 0.79 millimeter.

Apparatus

Two ultrahigh vacuum systems were used in this investigation. An apparatus capable of measuring adhesion, load, and friction was mounted in one of the vacuum systems (see fig. 1), which also contained tools for surface analysis (e.g., an Auger electron spectrometer). A gimbal-mounted beam is projected into the vacuum chamber. The beam contains two flats machined normal to each other with strain gages mounted thereon. The iron metal pin is mounted on the end of the beam. The load is applied by moving the beam normal to the flat and is sensed by a strain gage. The vertical sliding motion of the pin along the flat surface is accomplished through a motorized gimbal assembly. Under an applied load, the friction force is measured during vertical translation by the strain gage mounted normal to that used to measure load. This feature was used to examine the coefficient of friction at various loads.

The second ultrahigh vacuum system contained an XPS and is shown schematically in figure 2. The

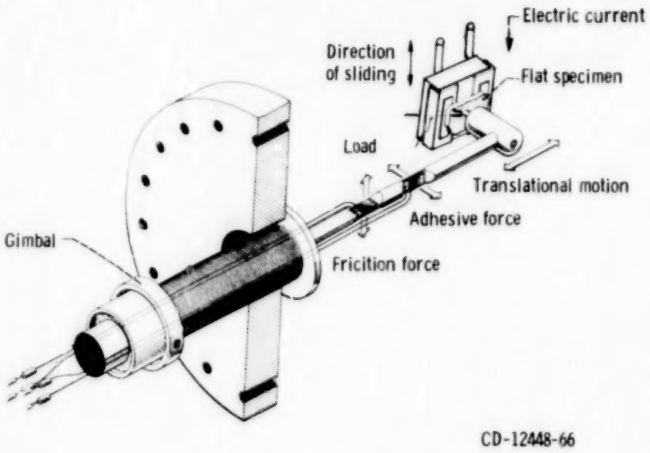


Figure 1. - High-vacuum friction and wear apparatus.

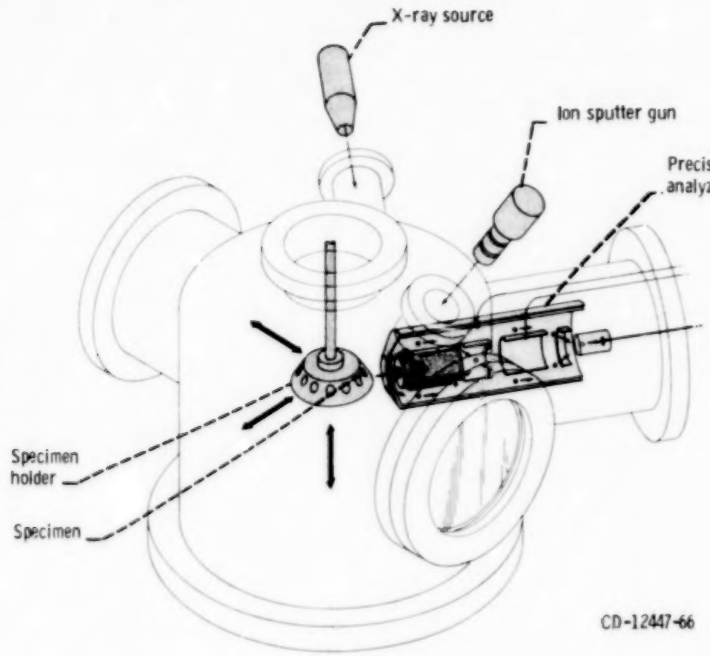
figure indicates the major components, including the electron energy analyzer, the X-ray source, and the ion gun used for ion sputter-etching. The X-ray source contains a magnesium anode. The specimens are mounted on the end of the specimen probe at an angle of 60° from the analyzer axis. The X-ray source is located at an angle of 79° from the analyzer axis.

Experimental Procedure

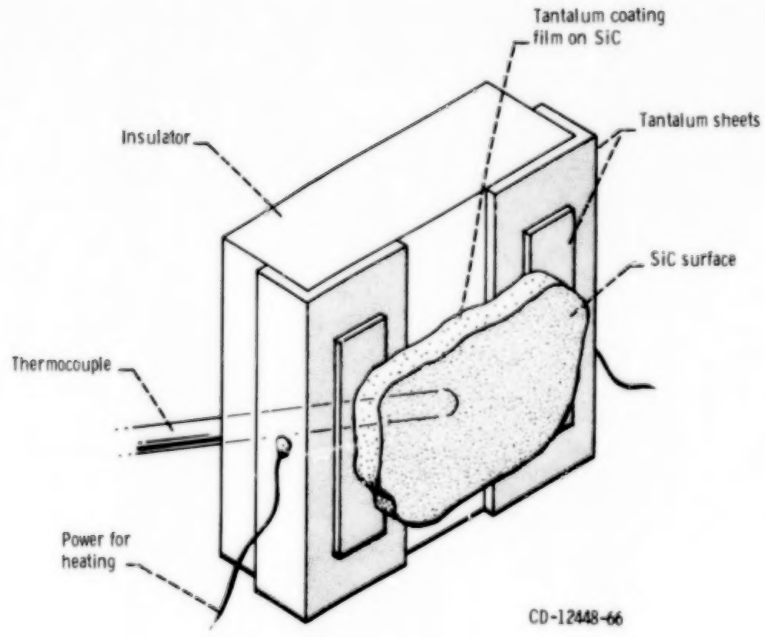
Specimen Preparation and Heating

To heat silicon carbide specimens by resistance heating, a tantalum thin film coating was applied to the rear surfaces of the silicon carbide crystals in a commercial radiofrequency diode sputtering apparatus. After coating, the silicon carbide surfaces to be analyzed were polished and cleaned, first with a diamond powder (3 μm diam.) and then with aluminum oxide powder (1 μm diam.). The back sides of specimens containing the tantalum films were attached to the tantalum rods or sheets with tantalum supporting sheets (figs. 1 and 2). The tantalum coating of the specimen was directly in contact with the rods or sheets. The flat and pin surfaces were rinsed with 200-proof ethyl alcohol just before they were placed in the vacuum chamber. The specimens were placed in the vacuum chamber (as shown in figs. 1 and 2), and the system was evacuated and subsequently baked at 250° C to obtain a pressure of 10^{-8} pascal (10^{-10} torr).

Once this pressure was obtained, argon gas was bled back into the vacuum chamber to a pressure of 1.3 pascal, and the pin specimen was argon-ion



(a) Spectrometer.



(b) Specimen holder.

Figure 2. - Schematic representations of the X-ray photoelectron spectrometer and silicon carbide specimen.

bombarded for 30 minutes at a −1000-volt direct-current potential. After this, the vacuum chamber was reevacuated.

Surface treatments were then conducted in situ on the silicon carbide flat specimens in both vacuum chambers. The surface treatments included heating to a maximum temperature of 1500° C at a pressure of 10^{-8} pascal and subsequent cooling to room temperature with the crystal in the as-received state. The surface of the silicon carbide was resistance-heated at various temperatures, starting at 250° C. The specimen was heated for periods of 1 or 3 hours

at a pressure of 10^{-8} pascal. After heating, the specimen was cooled to room temperature. AES or XPS spectra of the specimen were obtained before and after heating. Specimens were also heated to 400°, 500°, 700°, and 800° C, respectively. AES or XPS analyses after cooling to room temperature were conducted on these specimens in the same manner as those at 250° C. Heating times were for 1 or 3 hours at each temperature in a vacuum of 10^{-8} pascal.

The power for resistance-heating the silicon carbide specimen is supplied through the tantalum rods or sheets and the coated tantalum film by a precisely regulated direct-current output, adjustable over a wide range. The temperature of the silicon carbide surface was measured with a conventional thermocouple in direct contact with it.

Chemical Analyses of Surface

Recent articles have indicated a considerable interest in AES and XPS (refs. 7 and 8). The techniques are very useful in providing analyses of the first few atom layers of the surface of the specimens. The analysis depth with AES is on the order of 10 Å or less, and elemental concentrations as low as 0.1 percent of a monolayer can be detected and identified. The analysis depth with XPS is of the order of 20 Å or less, and the ultimate sensitivity is sufficient to allow fractions of a monolayer to be detected and identified.

A number of applications of AES and XPS as analytical surface tools in the study of tribology have been made (refs. 9 to 11). Both qualitative and quantitative information can be obtained with AES and XPS, for all elements in the periodic table above helium and adjacent elements are clearly distinguished. With hydrogen and helium, there are not enough occupied energy levels for detection of these elements. The measurements were conducted in the vacuum systems (10^{-8} Pa). To evaluate charging effects and to correct for electron energy shifts, spectra were recorded several times.

X-ray Photoelectron Spectroscopy

To obtain reproducible results, a strict standardization of the order and time of recording was used. The instrument was regularly calibrated. The analyzer calibration was determined by assuming the binding energy for the gold $4f_{7/2}$ peak to be 83.8 electron volts (eV); that is, the Au $4f_{7/2}$ level was used as the reference line. All survey spectra, scans of 1050 or 1100 eV, were taken at a pass energy of 50 or 100 eV, providing an instrumentation resolution of 1 eV at room temperature. The Mg Kα X-ray were used with an X-ray source power of 400 watts (10 kV, 40 mA). The narrow scans of the C_{1s}, Si_{2p} and O_{1s} are just wide enough to encompass the peaks of interest and were obtained with a pass energy of 25 eV at room temperature.

To evaluate the charging effect and to determine accurately the energy and shape of peaks, spectra were recorded several times. Resolution of the spectral peak is 1.5 eV, full width. The energy resolution is 2 percent of the pass energy, that is, 0.5 eV. The peak maxima can be located to ±0.1 eV. The reproducibility of peak height was good, and the probable error in the peak heights ranged from ±2 percent to ±8 percent. Peak ratios were generally good to ±10 percent or less.

Friction Experiments

In situ friction experiments were conducted with the surface-treated silicon carbide specimens over a temperature range of from room to 1200° C instead of 1500° C in order to avoid the iron melt. A load of 0.2 newton was applied to the pin-flat contact by deflecting the beam of figure 1. To obtain consistent experimental conditions, the time in contact before sliding was 30 seconds. Both the load and friction force were continuously monitored during a friction experiment. Sliding velocity was 3×10^{-3} meter per minute with a total sliding distance of 2 to 3 millimeters. All friction experiments were conducted in a 10^{-8} pascal vacuum. The coefficients of friction reported herein were obtained by averaging 3 to 5 measurements. The standard deviations of the data are within 2 to 4 percent of the average value.

Results and Discussion

Auger Electron Spectroscopy

An Auger electron spectrum of the single-crystal silicon carbide (0001) plane obtained before heating is presented in figure 3. The crystal was in the as-received state after it had been baked out in the vacuum system of 10^{-8} pascal. A carbon peak due to contamination was evident as well as an oxygen peak. The spectrum was similar to that obtained for amorphous-carbon (ref. 12 and 13). Three characteristic peaks for carbon, which are observed in the data of this paper, will be described later.

The spectra of the heated surfaces at 250°, 400°, and 600° C (fig. 4) reveal that the silicon carbide surfaces contained impurities, such as sulfur, oxygen, nitrogen, etc. Auger peaks for carbon, however, indicate a carbide-type carbon peak.

The spectrum of the surface heated to 800° C (fig. 5) is the same as that obtained for an argon sputter-cleaned surface presented in references 4 and 12: Contamination peaks have disappeared from the

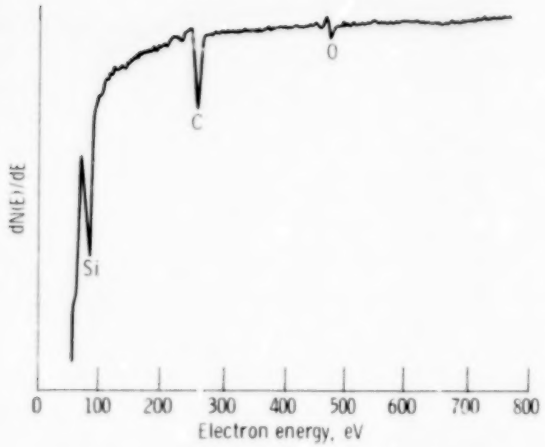


Figure 3. - Auger spectrum of silicon carbide (0001) surface after bake out at 250° C.

spectrum; and, in addition to the silicon peak, Auger peaks indicate carbide-type carbon on the surface of silicon carbide.

The spectra of the surface heated to 1000°, 1200°, and 1500° C (fig. 6) clearly reveal two characteristics: (1) a graphite-type carbon peak at 271 eV and (2) a decrease of silicon peak with increasing temperature. The carbide-type carbon peak changed to the graphite type at 900° to 1000° C, while the silicon peak decreased in intensity. The decrease of silicon peak intensity is due to preferential evaporation of silicon from the silicon carbide. The mechanism for graphite formation is that two successive carbon layers on the surface of silicon carbide, after evaporation of silicon, collapse into one layer of carbon hexagons with the unit mesh parallel to that of silicon carbide (refs. 13 and 14).

Figure 7 shows the typical Auger carbide-type, graphite-type, and amorphous carbon peaks. The carbide-type peaks are characterized by three peaks labelled A_0, A_1, and A_2 in figure 7(a), where A is used to denote an Auger peak. The graphite form is characterized by a step, where the position is labelled A in figure 7(b). Amorphous carbon appears only as the single main carbon peak labelled A in figure 7(c).

In figure 8 the relative heights of the carbon peaks and of silicon peaks are shown as a function of temperature. The relative peak heights increase with increasing temperature to 500° C. It decreases, however, with an increase of temperature in the range of 500° C to 800° C. Above 800° C it increases rapidly with increasing temperature and it approaches infinity at 1500° C.

X-ray Photoelectron Spectra

The XPS spectra of the C_{1s} and Si_{2p} obtained from narrow scans on the single-crystal silicon carbide surface are presented in figures 9 and 10. The as-

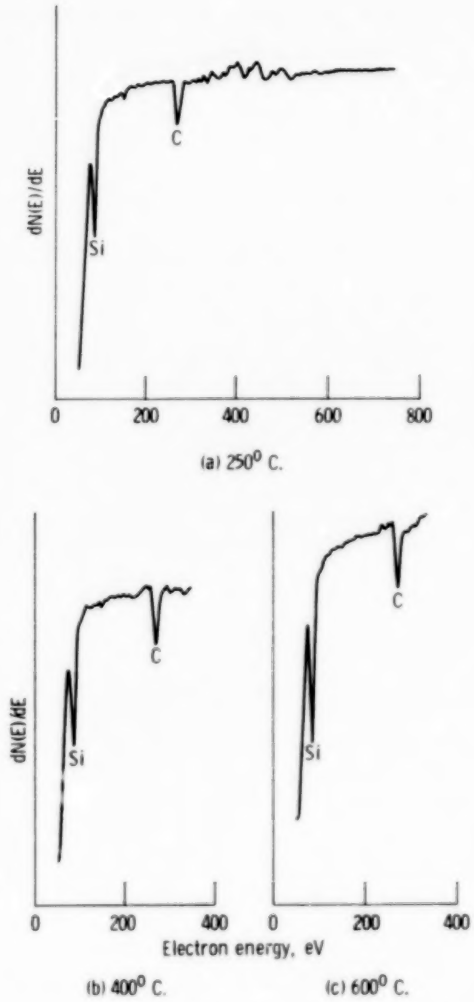


Figure 4. - Auger spectra of silicon carbide surface after heating at 250°, 400°, and 600° C.

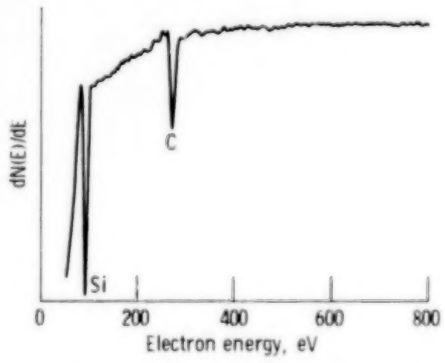


Figure 5. - Auger spectrum of silicon carbide (0001) surface after heating at 800° C.

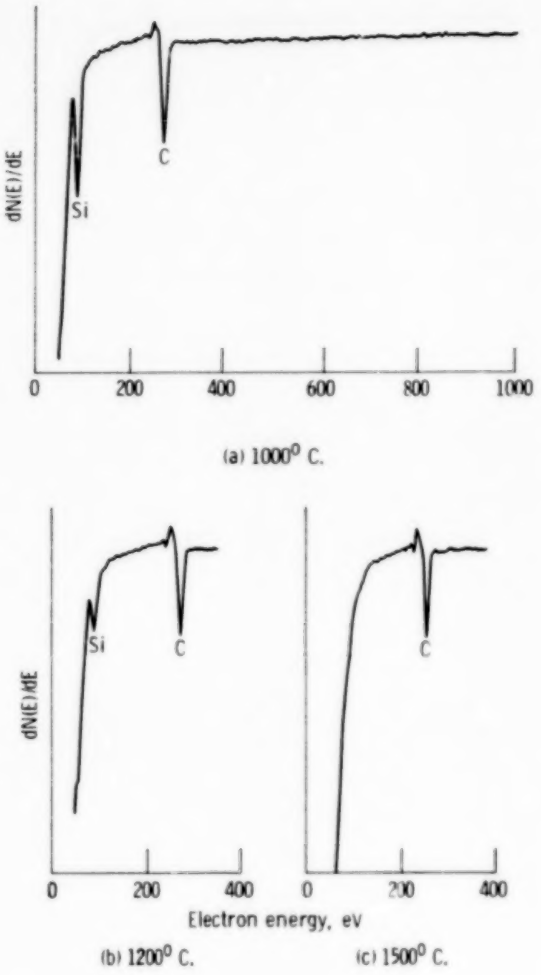


Figure 6. - Auger spectra of a silicon carbide surface after heating at 1000°, 1200°, and 1500° C.

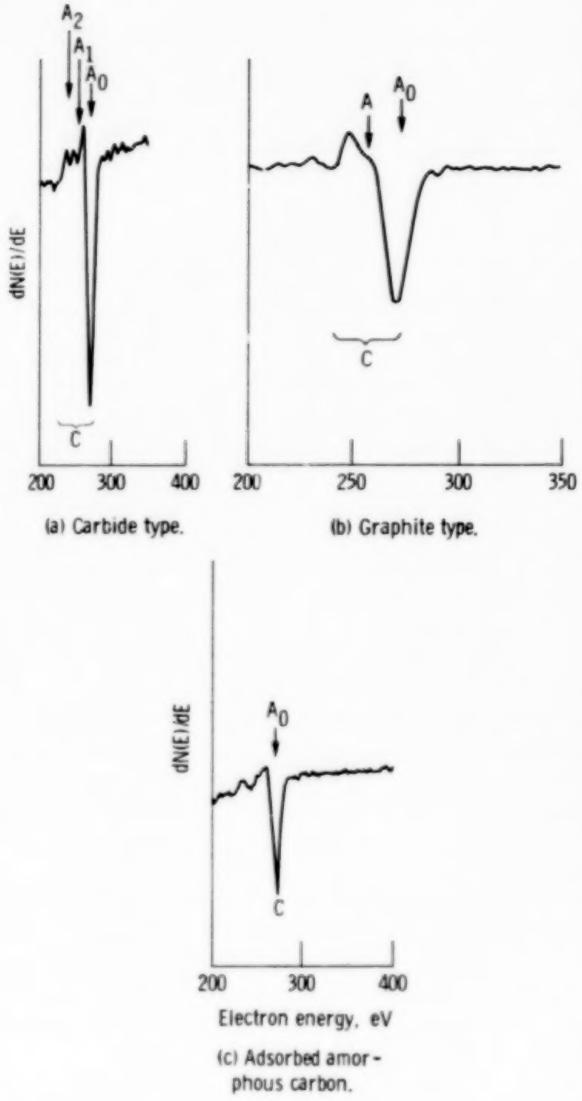


Figure 7. - Auger peaks of carbon on a silicon carbide (0001) surface.

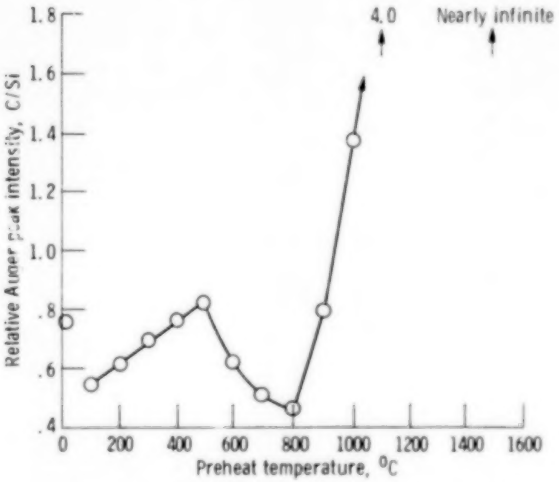


Figure 8. - Ratio of carbon to silicon Auger peak height in the silicon carbide (0001) surfaces preheated at various temperature.

received crystal after bake out was preheated at various temperatures in a 10^{-8} pascal vacuum. Preheating temperatures given in figures 9 and 10 are the highest temperatures to which the crystal had been heated. All the XPS spectra were taken at room temperature after bake out and preheating. The Si_{2p} photoelectron peak energies associated with silicon carbide at the various temperatures undergo a gradual change with an increase of preheating temperature from 100.4 eV (at the bake out temperature of 250° C) to 100.0 eV (at 1500° C preheating temperature). The vertical height, peak to base line, of the Si_{2p} peak in the spectra were minimum at the bake out temperatures and were the highest at 800° C. They were nearly the same at the temperatures of 400° C and 600° C. But above 800° C the Si_{2p} for silicon carbide decreased gradually with increasing temperature.

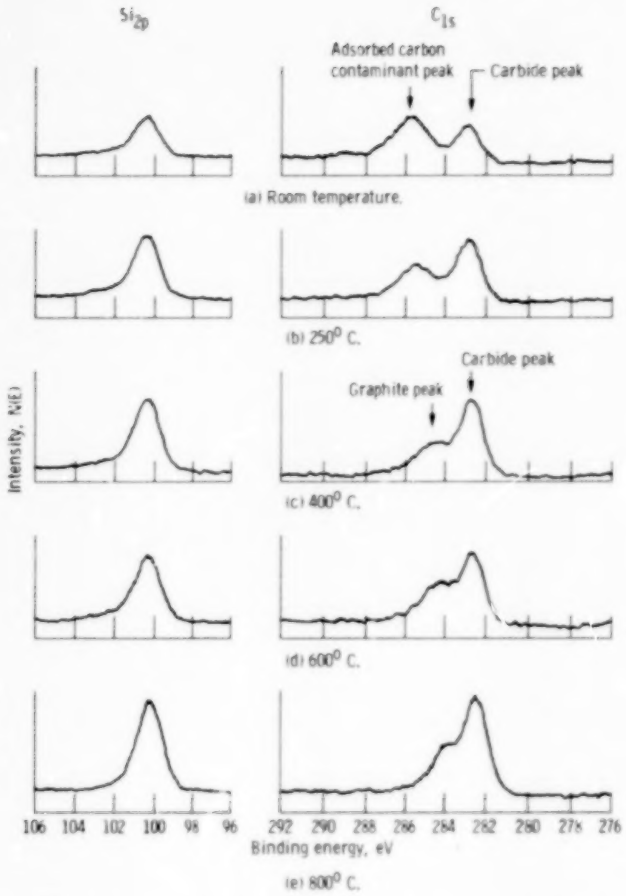


Figure 9. - Representative Si_{2p} and C_{1s} XPS peaks on silicon carbide (0001) surface preheated at various temperatures to 800° C.

Photoelectron lines for C_{1s} of the silicon carbide are split asymmetrically into doublet peaks (figs. 9 and 10). The results show a significant temperature influence on the silicon carbide surface. Three spectral features, which are dependent on the chemical nature of the specimen, are observed: (1) two kinds of doublet peaks, (2) change of the vertical height of the peaks, and (3) shift of peaks.

The doublet peaks are due to distinguishable kinds of carbon, that is, (1) a carbon contamination peak and a carbide peak at the room temperature, and (2) the graphite and the carbide peaks at temperatures from 400° to 1500° C. The XPS spectra of the as-received are shownin figure 9(a). The peak height of the carbon contamination is higher than that of carbide. At this temperature, the primary peaks are the adsorbed amorphous carbon contamination and carbide, and the contaminant peak height is lower than that of the carbide. For specimens preheated to 400° C the carbon contamination peak disappears from the spectrum. The graphite and carbide peaks are seen in the spectra of the specimens preheated to 400° C and above. Both the peak heights of graphite and the carbide are increased with an increase of

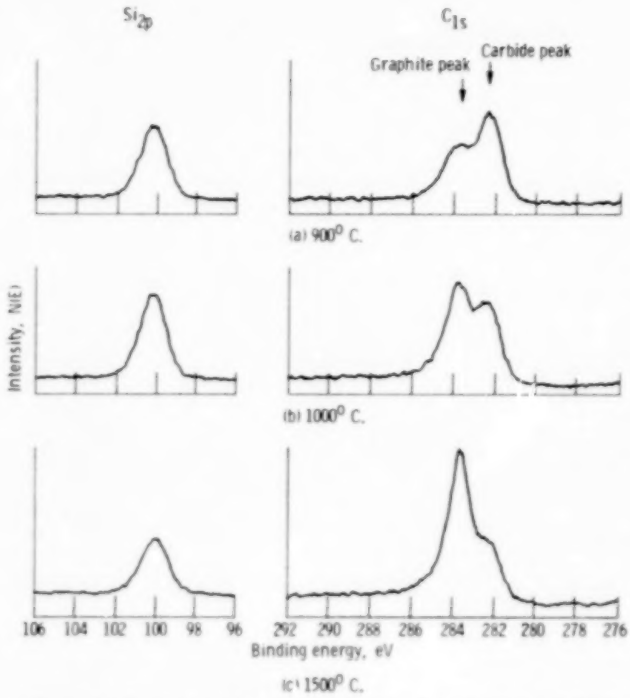


Figure 10. - Representative Si_{2p} and C_{1s} XPS peaks of the silicon carbide (0001) surface preheated at various temperatures from 900° to 1500° C.

preheating temperatures. A large carbide peak is clearly distinguished at a preheat temperature of 800° C.

In the previous section AES analysis of silicon carbide preheated at 800° C indicated that (1) the AES spectrum includes carbide-type carbon as well as a silicon peak on the surface. The spectrum indicates that the surface is pure silicon carbide. But XPS analysis, which can provide more detailed chemical information than AES, clearly indicates the presence of graphite on the silicon carbide surface preheated to 800° C.

At 900° C the carbide peak height was smaller than that at 800° C, but the graphite peak height was larger. The carbide peak height, however, was still larger than that of the graphite. At 1000° C, however, the height of the carbide peak decreased and became smaller than that of the graphite. Further, at 1500° C the height of the carbide peak becomes very small or nonexistent, while the graphite peak is very large.

In the previous section AES analysis of silicon carbide preheated at 1500° C (fig. 6(c)) indicated that the silicon AES peak had almost disappeared and that the carbon peak shown was only the graphite form.But XPS analysis (fig. 9) clearly indicated the evidence for silicon and carbide being present as well as graphite on the silicon carbide surface preheated to 1500° C. This difference can be accounted for in the fact that XPS analysis depth is deeper and is more sensitive to the presence of silicon than is AES.

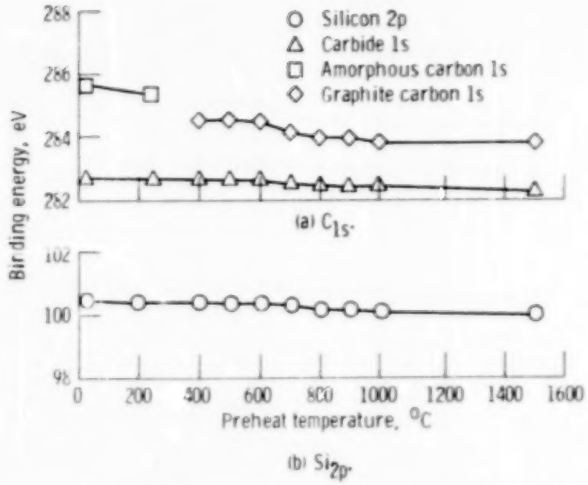


Figure 11. - Dependence of binding energies of Si_{2p} and C_{1s} levels of silicon carbide (0001) surface on temperature.

The third characteristic, that is, the shift of C_{1s} and Si_{2p} peaks on the silicon carbide surface, is presented in figure 11. The positions are determined from the narrow scans as shown in figures 9 and 10.

All the C_{1s} of carbon, carbide, and graphite and the Si_{2p} of silicon carbide are shifted toward lower binding energy with the increase of temperature. The carbon contamination peak, C_{1s} at 285.6 eV for the bake out temperature, is shifted toward a lower binding energy with a temperature rise. The shift is small (0.3 to 0.4 eV) and is almost the same as the instrumental error (±0.3 eV). The shift of the carbide peak is in the range of 282.7 eV at the bake out temperature to 282.2 eV at 1500° C, and that of graphite is 284.5 eV at 400° C to 283.6 eV at 1500° C. The shift of Si_{2p} is from 100.5 eV at the bake out temperature to 100.0 eV at 1500° C.

The heights of the Si_{2p} and C_{1s} (adsorbed carbon, graphite, and carbide) peaks on the silicon carbide surface are plotted as functions of temperature in figure 12. There are distinct influences of temperature on Si_{2p} and C_{1s} concentrations. The trend of Si_{2p} is similar to that of the carbide C_{1s} up to 1000° C. The graphite C_{1s} height increases rapidly with increasing temperature above 900° C. In figure 13 the relative heights of the adsorbed carbon, graphite, and carbide C_{1s} peaks to that of Si_{2p} peak are shown as a function of temperature. The relative carbon C_{1s} peak height decreases rapidly with increasing temperature to 400° C. The relative graphite – and carbide – C_{1s} peak heights are relatively constant below 900° C, but above 900° C the graphite C_{1s}/Si_{2p} increases rapidly with increasing temperature, while the carbide C_{1s}/Si_{2p} decreases at 1000° C and then levels off with further increases in temperature.

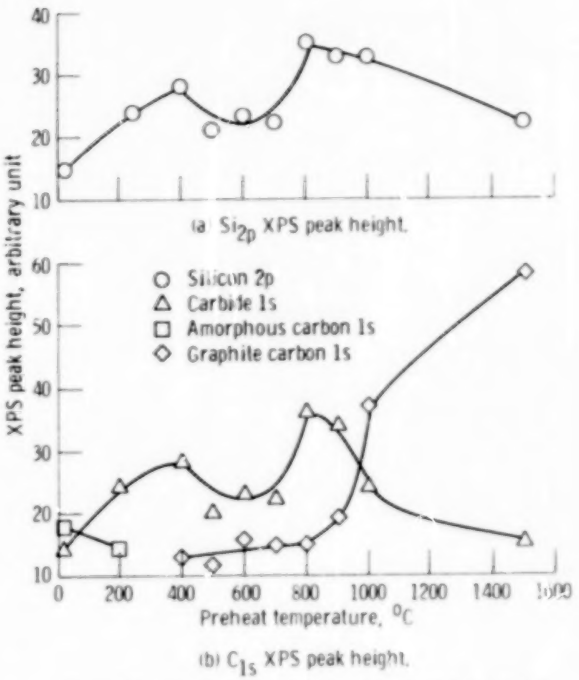


Figure 12. - Variations of Si_{2p} and C_{1s} peak heights for preheated silicon carbide (0001) surfaces.

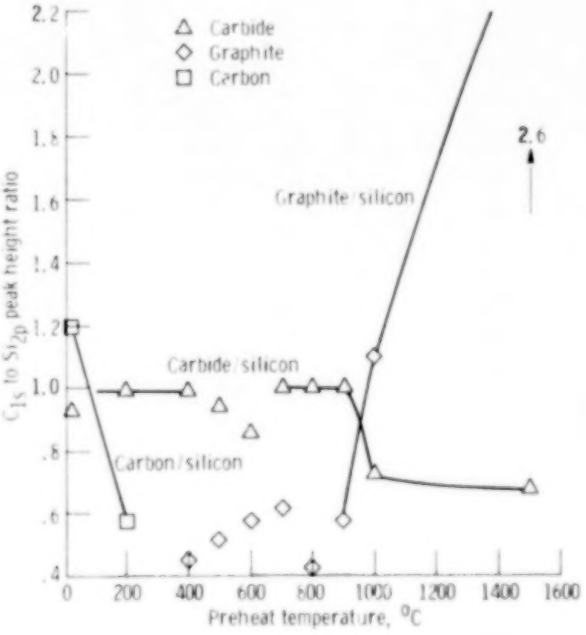


Figure 13. - Ratios of C_{1s} to Si_{2p} XPS peak heights on the silicon carbide (0001) surfaces preheated at various temperatures.

Friction Behavior

Sliding friction experiments were conducted with single-crystal silicon carbide in contact with iron in vacuum. Friction-force traces resulting from such sliding are generally characterized by a stick-slip

behavior as is shown representatively in figure 14. All the coefficients of friction reported in figures 14 to 16 are static. The coefficient of static friction μ is defined as $\mu = F_1/W$, where F_1 is the friction force at which the first break, that is, first motion, is observed in the friction-force trace and W is the normal load. The kinetic friction properties of silicon carbide in sliding contact with iron were generally of the same magnitude as those of the static friction. The kinetic friction is also defined as $\mu = F/W$, where F is the friction force determined by averaging the heights of maximum peaks in the friction-force trace, and W is the normal load.

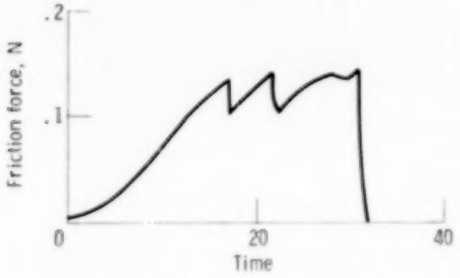


Figure 14. - Friction force trace for an iron rider sliding on silicon carbide (0001) in the $\langle 11\bar{2}0 \rangle$ direction surface. Normal load, 0.2 N; sliding temperature, 800° C; vacuum, 10^{-8} Pa.

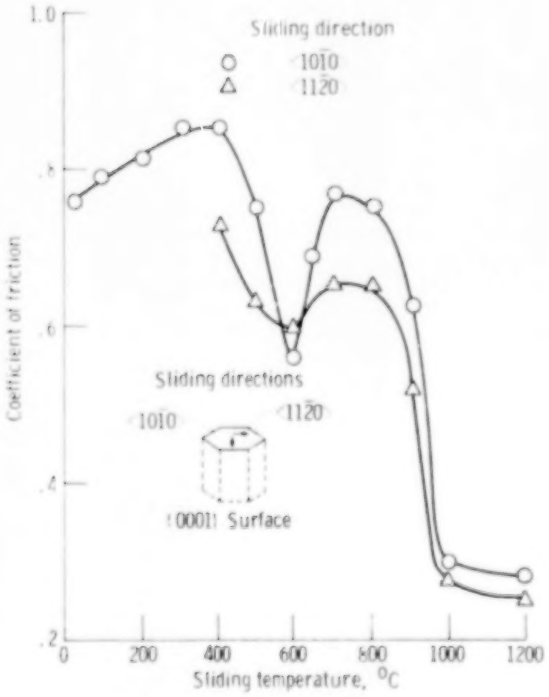


Figure 15. - Effect of temperature on coefficient of friction for silicon carbide (0001) surface sliding against an iron rider. The iron rider was argon ion sputter cleaned before experiments. Normal load, 0.2 N; vacuum, 10^{-8} Pa.

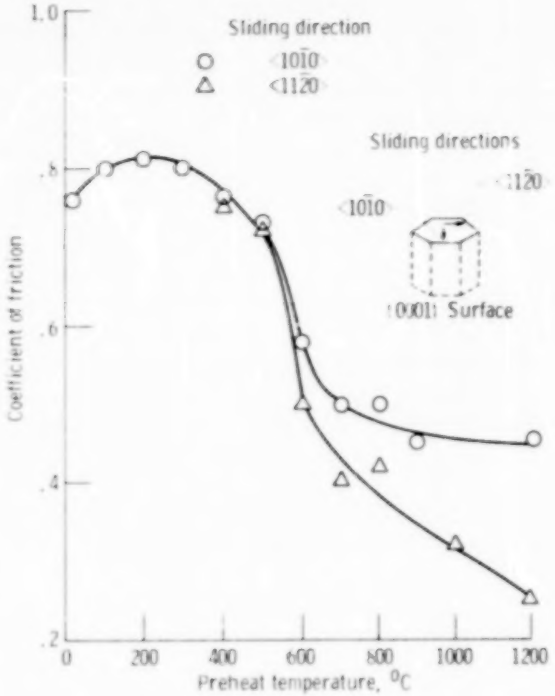


Figure 16. - Effect of preheating temperature on coefficient of friction for silicon carbide (0001) surface sliding against an iron rider at room temperature. The silicon carbide surfaces were preheated and the iron was argon ion sputter cleaned before experiments. Normal load, 0.2 N; vacuum, 10^{-8} Pa.

The coefficient of friction of the silicon carbide (0001) surfaces in contact with iron as a function of sliding temperatures is indicated in the graph of figure 15. The iron rider was sputter-cleaned with argon ions. The silicon carbide specimen was first baked out in the vacuum system, and then heated to the sliding temperature. The coefficient of friction increased slightly with increasing sliding temperatures below 400° C; decreased with an increase in temperatures from 400° to 600° C; and increased with sliding temperatures from 600° to 800° C. The general decrease in friction at 400° to 600° C is due to the gradual removal of the contaminants of carbon and oxygen from the surface. And the increase in friction at 600° to 800° C is associated with increased adhesion and plastic flow in the contact area. Above 800° C the coefficient of friction decreases rapidly with an increase of temperature. The rapid decrease in friction above 800° C correlated with the graphitization of the silicon carbide surface already discussed in previous sections. It is interesting to note that there is a similarity between the trends of friction with increasing temperature shown in figure 15, and the Si_{2p} and the carbide C_{1s} XPS peak heights shown in figure 12.

The coefficients of friction of silicon carbide (0001) surfaces in contact with iron at room temperature are presented in figure 16 for various preheat temperatures up to 1200° C. The silicon carbide surfaces were first preheated in a vacuum of 10^{-8} pascal. The coefficient of friction increases slightly with increasing sliding temperature below 200° C. Above 400° C the coefficient of friction decreases with an increase in temperature. The general decrease in friction at these temperatures may be mainly due to gradual removal of carbon and oxygen contamination. The graphite on the silicon carbide surface above 400° C, which appears in the XPS analysis, correlates again with the decrease in friction.

Summary of Results

From the results of AES and XPS analyses and sliding friction experiments conducted in this investigation with single crystal silicon carbide (0001) surfaces heated to 1500° C, the following conclusions are drawn:

1. All the results represent a significant temperature influence on the surface and friction properties of silicon carbide.

2. The coefficients of friction of polycrystalline iron sliding against single-crystal silicon carbide (0001) surfaces were high at temperatures to 800° C. Similar coefficients of friction were obtained at room temperature when the silicon carbide was preheated at various temperatures to 800° C. For friction experiments conducted above 800° C or for specimens preheated above 800° C, the coefficients of friction were dramatically lower.

3. The main contamination on the silicon carbide surfaces is amorphous carbon, which disappears on heating to 400° C. Above 400° C graphite and carbide are primarily seen, in addition to silicon. At 800° C the silicon and carbide-type carbon are at maximum intensity as determined by the XPS and AES spectra. Above 800° C then graphite increases rapidly with an increase in temperature, while the carbide-type carbon and silicon decrease rapidly in intensity.

4. The physical conclusion from the surface analysis is that the surface of silicon carbide graphitizes.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, October 10, 1980

References

1. O'Connor, J. R.; and Smiltens, J., eds.: Silicon Carbide, a High Semiconductor. Pergamon Press, Inc., 1960.
2. Marshall, R. C.; Faust, J. W., Jr.; and Ryan, C. E., eds.: Silicon Carbide – 1973. University of South Carolina Press, 1974.
3. Komanduri, R.; and Shaw, M. C.: Attritious Wear of Silicon Carbide. J. Eng., Ind., vol. 98, no. 4, Nov. 1976, pp. 1125-1134.
4. Miyoshi, K.; and Buckley, Donald H.: Changes in Surface Chemistry of the Silicon Carbide (0001) Surface with Temperature and Their Effect on Friction Behavior. NASA TP-1756, 1980.
5. Taylor, A.; and Laidler, D. S.: The Formation and Crystal Structure of Silicon Carbide. Br. J. Appl. Phys., vol. 1, no. 7, July 1950, pp. 174-181.
6. Hume-Rothery, W.; and Raynor, G. V.: The Structure of Metals and Alloys. 4th, ed. rev. The Institute of Metals. (London), 1962.
7. Swingle, Robert S., II; and Riggs, W. M.: ESCA Crit. Rev. Anal. Chem., vol. 5, no. 3, Oct. 1975, pp. 267-321.
8. Kane, Philip F.; and Larrabee, Craydon B., eds.: Characterization of Solid Surfaces. Plenum Press, 1974.
9. Buckley, Donald H.: The Use of Analytical Surface Tools in the Fundamental Study of Wear. Wear, vol. 46, 1978, pp. 19-53.
10. Wheeler, Donald R.: X-ray Photoelectron Spectroscopic Study of Surface Chemistry of Didenzyl Disulfide on Steel Under Mild and Severe Wear Conditions. Wear, vol. 47, Apr. 1978, pp. 243-254.
11. Ferrante, John: Practical Applications of Surface Analytic Tools in Tribology. NASA TM-81484, 1980.
12. Miyoshi, K.; and Buckley, D. H.: Effect of Oxygen and Nitrogen Interactions on Friction of Single-Crystal Silicon Carbide. NASA TP-1265, 1978.
13. Badami, D. V.: Carbon, X-ray Studies of Graphite Formed by Decomposing Silicon Carbide. Vol. 3, no. 1, 1965, pp. 53-57.
14. Van Bommel, A. J.; Crombeen, J. E.; and Van Tooren, A.: LEED and Auger Electron Observations of the SiC (0001) Surface. Surf. sci., vol. 48, 1975, pp. 463-472.

1. Report No. NASA TP-1813	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle SURFACE CHEMISTRY AND FRICTION BEHAVIOR OF THE SILICON CARBIDE (0001) SURFACE AT TEMPERATURES TO 1500° C		5. Report Date March 1981 6. Performing Organization Code 506-53-12
7. Author(s) Kazuhisa Miyoshi and Donald H. Buckley		8. Performing Organization Report No. E-542 10. Work Unit No.
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135		11. Contract or Grant No. 13. Type of Report and Period Covered Technical Paper
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

X-ray photoelectron and Auger electron spectroscopy analyses and friction studies were conducted with a silicon carbide (0001) surface in contact with iron at various temperatures to 1200° or 1500° C in a vacuum of 10^{-8} Pa. The results indicate that there is a significant temperature influence on both the surface chemistry and friction properties of silicon carbide. The principal contaminant of adsorbed amorphous carbon on the silicon carbide surface in the as-received state is removed by simply heating to 400° C. Above 400° C, graphite and carbide type-carbon are the primary species on the silicon carbide surface, in addition to silicon. The coefficients of friction of polycrystalline iron sliding against a single-crystal silicon carbide (0001) surface were high at temperatures to 800° C. Similar coefficients of friction were obtained at room temperature after the silicon carbide had been preheated at various temperatures up to 800° C. When the friction experiments were conducted above 800° C or when the specimens had been preheated to above 800° C, the coefficients of friction were dramatically lower. At 800° C the silicon and carbide type-carbon are at a maximum intensity in the XPS spectra. With increasing temperature above 800° C, the concentration of the graphite increases rapidly on the surface, whereas those of the carbide type-carbon and silicon decrease rapidly.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Friction Silicon carbide Surfaces	Unclassified - unlimited STAR Category 27

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	12	A02

* For sale by the National Technical Information Service, Springfield, Virginia 22161

NASA-Langley, 1981

END

6-11-81